भारतीय मानक
Indian Standard

IS 7645 : 2021

फिनाइल जे-अम्ल, तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

Phenyl J-Acid, Technical — Specification

(Second Revision)

ICS 71.080.40

भारतीय मानक ब्यूरो
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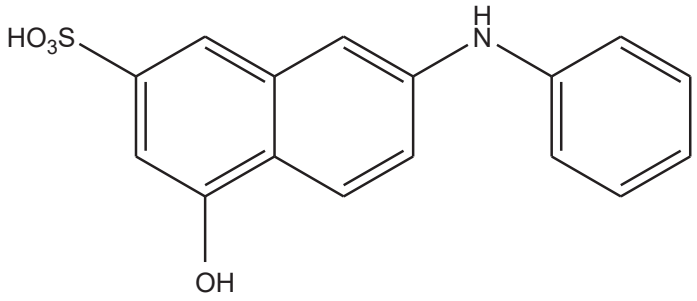
August 2021 **Price Group 4**

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by Dye Intermediates Sectional Committee had been approved by the Petroleum Coal and Related Products Division Council.

Phenyl J-acid, chemically described as 2-phenglamino-5-naphthol-7-sulphonic acid ($C_{16}H_{13}O_4NS$) is an important intermediate used in the manufacture of dyes. It is also known as 2-phenylamino-5-hydroxy naphthalene-7-sulphonic acid. It is represented by the following structural formula:



N-PHENYL-J-ACID
(MOLECULAR MASS 315)
CAS Registry Number [119-40-4]

This standard was first published in 1975 and subsequently revised in 1994. In the first revision, the requirements of matter insoluble in dilute sodium carbonate and J-acid content were modified.

Considering to the development in latest analytical techniques in the last 15 years, committee decided to revise this standard to incorporate purity by high performance liquid chromatography and impurities of aniline and J acid content in N-phenyl J acid by HPLC method.

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off-in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHENYL J-ACID, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for phenyl J-acid, technical.

2 REFERENCES

The following Indian Standards contain provisions which through reference in the text, constitute provisionsof this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
797 : 1982	Common salt for chemical industries (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a paste, or, if dry, in the form of light grey to greenish grey powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

4.2 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of material;
d) Month and year of the manufacture;
e) Batch or lot number; and
f) The minimum cautionary notice worded.

"IT IS A MILD SENSITIZER LOCAL CONTACT MAY CAUSE DERMATITIS"

Table 1 Requirements for Phenyl J-Acid, Technical

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity by Coupling Value, *Min*	85	A
ii)	Purity by HPLC (on dry basis)	85	B
iii)	Aniline content, percent by mass (HPLC method), *Max*	0.2	C
iv)	J-acid content, percent by mass (HPLC method), *Max*	0.5	C
v)	Matter insoluble in dilute sodium carbonate solution, percent by mass, *Max*	0.3	**11.2** of IS 5299

4.2.1 The containers may also be marked with the Standard Mark.

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Numbers of Tests

5.2.1 Tests for assay shall be conducted on each of the individual samples separately.

5.2.2 Tests for the determination of the remaining characteristics, namely, aniline content, J-acid content and matter insoluble in dilute sodium carbonate solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

The lot shall be declared as conforming to the requirements of aniline content, J-acid content and matter insoluble in dilute sodium carbonate solution if the test results satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to methods prescribed in Annexes and IS Specifications as given in column 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No*. (i)]

DETERMINATION OF PURITY BY COUPLING VALUE METHOD

A-0 OUTLINE OF METHOD

The material is dissolved in dilute sodium carbonate solution. A known volume of the solution is coupled with standard 4-nitroaniline diazo in sodium carbonate and dilute sodium acetate medium and from the consumption.

A-1 PREPARED SAMPLE

Dry the material at 105 ± 1 °C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-2 REAGENTS

A-2.1 Alkaline H-Acid Indicator Solution

A-2.2 Sodium Acetate Solution — 20 percent (m/v).

A-2.3 Sodium Carbonate Solution — 10 percent (m/v).

A-2.4 4-nitroaniline Diazo Solution — 0.1 N.

A-2.5 Sodium Nitrite 1.0 N Solution

A-2.6 Common Salt — *see* IS 797.

A-2.7 Preparation of the PNA-Diazo Solution 0.1 mol/l (0.1 N)

6.9066 g of *p*-nitroaniline are weighed into a 400 ml glass beaker and dissolved in 100 ml of purify water and 50 ml of hydrochloric acid 30-32 percent. The solution is warmed and stirred until all compounds are completely dissolved. After, the solution is cooled down with ice to 3-5 °C. At this point, 50.00 ml of sodium nitrite, concentration ($NaNO_2$) = 1.0 mol/l are added directly into the solution in order to avoid a loss of NO_2 (NOx vapours.). The solution is poured into a 500 ml measuring flask and taken to volume with purify water. Keep the solution in ice bath.

A-2.8 Standardization of 0.1 N PNA Diazo Solution:- Weight 1.0 g (accuracy: 0.0001 g) β-naphthol and transfer in 500 ml beaker, add 10-15 ml of 1n NaOH to dissolved. Stir well if not dissolved. Add 200 ml distilled water . After addition of 30 ml sodium acetate solution, and add ice to cool it ,the solution is titrated gradually to the end point with p-nitro aniline-diazo (PNA-diazo) . Test with alkaline H-acid for excess diazo and with diazo for the coupling component. A faint test line with alkaline H-acid reproducible for a period of 5 min denotes the end point.

A-2.9 Calculation

Normality of PNA-diazo solution: -

$$\frac{99 \times \text{weight of } \beta\text{-Naphthol [STD] (in gm)}}{V \times M}$$

where

V = volume in ml, of the PNA-diazo solution used; and

M = mass in g, of β-naphthol taken for the test.

A-3 PROCEDURE

Weight accurately about 1 g of the prepared sample (*see* **A-l)** and transfer to a 500 ml beaker with the help of water. Add 20 ml of sodium carbonate solution and 100 ml of sodium acetate solution. Stir mechanically and cool with ice to 0 to 5 °C.Titrate with standard 4 nitroaniline diazo solution, from a cold water-jacketted burette. Add 15 g of common salt near the end point. Test with alkaline H-acid for excess diazo and with diazo for the coupling component. A faint test line with alkaline H-acid reproducible for a period of 5 min denotes the end point.

A-4 CALCULATION

Assay, percent by mass, on dry basis = $\frac{V \times N \times 31.5}{M}$

where

V = Volume in ml, of the standard 4 nitroaniline diazo solution used;

N = Normality of 4 nitroaniline diazo solution; and

M = Mass in g, of the prepared sample taken for the test.

ANNEX B

[*Table* 1, *Sl No*. (ii)]

PURITY BY HPLC

B-0 OUTLINE OF METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-1 OBJECTIVE

To determine purity of phenyl J-acid by high performance liquid chromatography.

B-2 APPARATUS

Binary gradient liquid chromatography system with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-3 COLUMN

Kinetex C18 100A 250 × 4.6 mm, 5 µm or equivalent.

B-4 REAGENTS

B-4.1 Acetonitrile, HPLC Grade

B-4.2 Water, HPLC Grade

B-4.3 Di-sodium Hydrogen Orthophosphate, HPLC Grade

B-4.4 Ammonium Di-hydrogen Orthophosphate, HPLC Grade

B-4.5 Phenyl J-acid, Known Purity

B-5 STANDARD PREPARATION

Weigh accurately 0.0500 gm standard phenyl J-acid in 100 ml volumetric flask dissolve it in water: acetonitrile (20 : 80) and make up to the mark with water: acetonitrile (20 : 80).

B-6 SAMPLE PREPARATION

Weigh accurately 0.0500 gm sample in 100 ml volumetric flask dissolve it in water: acetonitrile (20 : 80) and make up to the mark with water: acetonitrile (20 : 80).

B-7 BUFFER PREPARATION

Take 0.5750 gm ammonium di-hydrogen orthophosphate and 0.7000 gm di-sodium hydrogen orthophosphate in1 liter volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water.

B-8 FLOW RATE: 1.00 ml/min

B-9 MOBILE PHASE:

Actonitrile	Buffer
25	75

B-10 COLUMN OVEN TEMPERATURE: 40 °C

B-11 INJECTION VOLUME: 5 µl

B-12 RUN TIME: 15 min

B-13 WAVE LENGTH: 230 nm

B-14 PEAK TIME: J-acid - 2.34 min
Aniline - 5.81 min
Phenyl J-acid - 8.35 min

B-15 CALCULATION

Calculate the peak area of individual constituent pertaining to phenyl J-acid on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure phenyl J-acid.

$$\text{Percent of phenyl J-acid} = \frac{A2 \times V1 \times W1 \times B2}{A1 \times V2 \times W2 \times B1} \times 100$$

where

A1 = Area of standard phenyl J-acid;
V1 = Injection volume of standard phenyl J-acid;
W1 = Weight of standard phenyl J-acid;
B1 = Total volume of standard phenyl J-acid;
A2 = Area of phenyl J-acid peak in sample;
V2 = Injection volume of sample;
W2 = Weight of sample;
B2 = Total volume of sample;

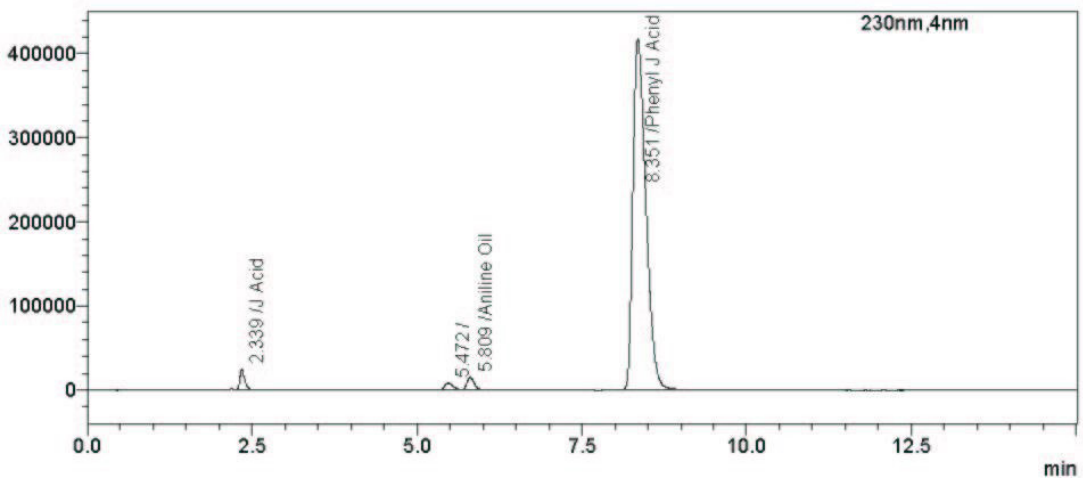


FIG. 1

ANNEX C

[*Table* 1, *Sl No.* (iii) *and* (iv)]

DETERMINATION OF J-ACID AND ANILINE CONTENT BY HPLC

C-1 OBJECTIVE

To determine J-acid and aniline content in N-phenyl J-acid by high performance liquid chromatography.

C-2 APPARATUS

Binary gradient liquid chromatography system capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-3 COLUMN

C18 100A 250 × 4.6 mm, 5 µm or equivalent.

C-4 REAGENT

C-4.1 Acetonitrile, HPLC Grade

C-4.2 Water, HPLC Grade

C-4.3 Di-sodium Hydrogen Orthophosphate, HPLC Grade

C-4.4 Ammonium Di-hydrogen Orthophosphate, HPLC Grade

C-4.5 Phenyl J-acid, Known Purity

C-5 STANDARD PREPARATION

C-5.1 Weigh accurately 0.0050 gm standard J-acid in 100 ml volumetric flask dissolve it in water: acetonitrile (20 : 80) and make up to the mark with water: acetonitrile (20 : 80). ---------- A

C-5.2 Take 1 ml of solution A dilute 10 ml with water : acetonitrile (20 : 80).

C-5.3 Weigh accurately 0.0050 gm standards aniline in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile. ---------- B

C-5.4 Take 1 ml of solution B dilute 10 ml with acetonitrile.

C-6 SAMPLE PREPARATION

Weigh accurately 0.0500 gm sample in 100 ml volumetric flask dissolve it in water: acetonitrile (20 : 80) and make up to the mark with water: acetonitrile (20 : 80).

C-7 BUFFER PREPARATION

Take 0.5750 gm ammonium di-hydrogen orthophosphate and 0.7000 gm di-sodium hydrogen orthophosphate in 1 litre volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water.

C-8 FLOW RATE: 1.00 ml/min

C-9 MOBILE PHASE:

Acetonitrile	Buffer
25	75

C-10 COLUMN OVEN TEMPERATURE: 40 °C

C-11 INJECTION VOLUME: 5 µl

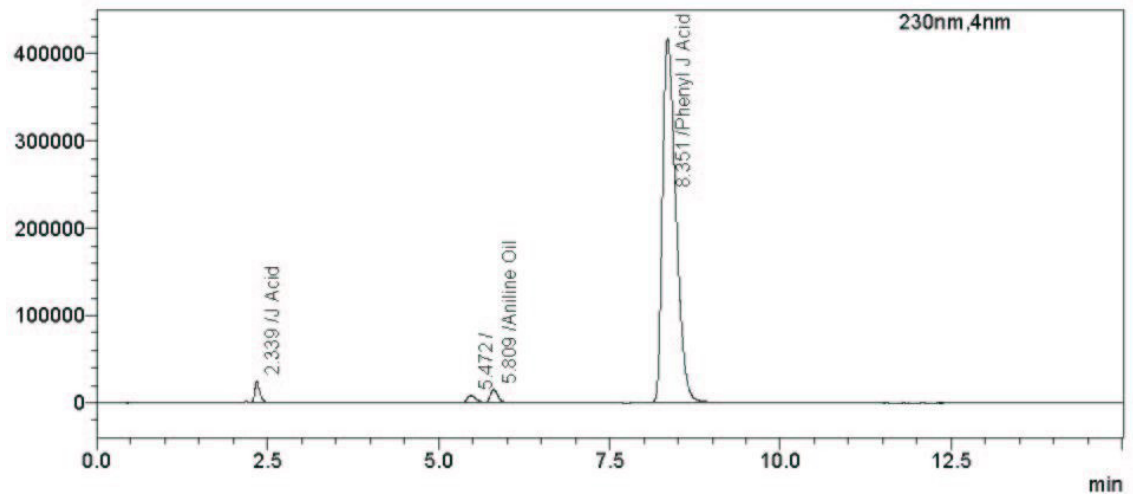


FIG. 2

C-12 RUN TIME: 15 min

C-13 WAVE LENGTH: 230 nm

C-14 PEAK TIME:

J Acid	- 34 min
Aniline	- 5.81 min
Phenyl J Acid	- 8.35 min

C-15 CALCULATION

Calculate the peak area of individual constituent pertaining to J-acid/aniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with standard J-acid/aniline.

Percent of J-acid/aniline =

$$\frac{A2 \times V1 \times W1 \times B2}{A1 \times V2 \times W2 \times B1} \times 100$$

where

A1	=	Area of standard J-acid/aniline;
V1	=	Injection volume of standard J-acid/aniline;
W1	=	Weight of standard J-acid/aniline;
B1	=	Total volume of standard J-acid/aniline;
A2	=	Area of Jacid/aniline peak in sample;
V2	=	Injection volume of sample;
W2	=	Weight of sample; and
B2	=	Total volume of sample.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee PCD 26

Organization	*Representative(s)*
Personal Capacity	Prof P. M. Bhate (***Chairman***)
Aarti Industries Limited, Mumbai	Shri Kirit H. Desai
Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat	Shri Mansukh H. Vekaria
Archroma, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited, Atul, Valsad, Gujarat	Dr J. G. Desai Dr M. U. Rahman (*Alternate*)
BASF India Limited, Mumbai	Shri Uday Kulkarni
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K. Astik (*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), Ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
Indian Chemical Council, Mumbai	Shri P. S. Singh
Jay Chemical Industries, Ahmedabad	Shri Vilpesh Yadav Shrimati Maitri Vyas (*Alternate*)
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Ministry of Defence (Research and Development), New Delhi	Dr Vijay Tak
National Test House (er), Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
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Organization	*Representative(s)*
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The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	Shri V. R. Kanetkar
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Member Secretary

Shri Chandrakesh Singh
Scientist 'D' (PCD), BIS

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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